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Impact of the Holding Time on the Micro structure of the T91 Steel at High Temperature

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Abstract: **The objective of this work is to characterize the evolution of the microstructure of the ASTM A213 T91 steel, used in power plants of electricity generation, for high temperatures up to 650° C, to optimize the performances of the installations and to reduce the pollutant emissions. The high temperature and pressure inside the boilers promote the microstructural evolution of the martensitic matrix of the T91 by increasing the size of the precipitates by the time exposure. The growth of these precipitates is explained by the absorption of the matrix elements, which will constitue in long term a favorable site for the nucleation of the microcavities; that triggers the creep phenomenon.**

An experience has been conducted in the aim of understanding the evolution of the microstructure ofthe steel at a temperature of 550° C. So far, four samples were removed from the furnance for time intervals of: 0, 260, 760 and 1260 hours. Examination by scanning electron microscopy of the martensitic matrix shows a slight increase of the sizes of precipitated M23C6 and stability for MX precipitates.

Keywords: **Microstructure, T91 Steel, ASTM A213.**

I. INTRODUCTION

The potential growth of the consumption of electrical energy worldwide and the universal challenges related to the Environmental Protection, are major constraints in the design of the electricity power generation. Indeed, new power plants should ensure long life time and respect for the environment. These challenges can not be assured without the development of steels that can withstand the high conditions of temperature and pressure. Various grades of steels have been developed in recent decades: the steels contained first about 2% of chromium (grades 21 and 22). Then, and since the 80th, steels with 9% of chromium (such as the steel of our study) has been developed, and recently steels with 12% chromium. Grades 91 and 92 have proven, since their integration in thermal power plants, high performance. The excellent mechanical properties, such as the low expansion coefficient and the high conductivity, make these steels prime candidates to work at high temperatures and pressures. The steel T91 is endowed with these properties which improve its creep strength and oxidation resistance.

The high performances of this steel are strongly related to its microstructure consisted of tempered martensite matrix rich in precipitates: the $M_{23}C_6$ are along the prior austenitic grain boundaries, the subgrains and laths, while the MX precipitates are finely distributed within laths. Nevertheless, this steel has served in a limited number of plants around the world since its integration. Therefore, its behavior for long periods stills the subject of many researchs. Exposure to creep for long periods causes changes in the microstructure of the steel, which affects the creep strength and also oxidation resistance.

To better characterize the behavior of the steel under operating conditions (temperature and pressure) and in function of time, we chose to start with the characterization of the material by simulating its operation in the installation by our laboratory means. Presumably start finding changes in a few thousand hours of turning on temperature (which must be sufficiently high: between 500 and 600° C).

The laboratory is equiped with furnances to expose different samples under a temperature of 550°C (without load) and the maximum time we can do: we have reached 1300 hours so far and we plan to reach 10,000 hours, to extrapolate any laws of evolution of certain parameters related to the microstructure: evolution of precipitates sizes, grain size, orientation lath martensite etc.

Second terms, the specimens will be exposed to creep (with load) in the creep machine to determine the impact of the load on the microstructural evolution.

For this, a creep machine has been in place for us to unwind creep tests according to international standards. For the characterization of the material, we use the following test methods: optical microscopy, scanning electron microscopy, EDS (energy dispersive X-ray spectrometry) and a Vickers machine.

II. THE STEELS USED IN POWER GENERATION

In this section, we will discuss the different families of steels used in thermal installations by historical.

1. Classification Of Heat Resistant Steels

The choice of thematerial depends on the load and the maximum temperature that must endure. Untill the 80th, the steels with 2% of chromium containt (2.25% of chromium and 1% of molybdenum) were frequently used, it resisted to temperatures of 400 to 500°C with loads up to 35 MPa. These performances are improved by adding vanadium, an element which contributes to a significant improvement in the creep strength of the steel, which makes up the served pressure to 60 MPa. Then, the development of steels with 9% chromium increases in a significant way the steam and pressure parameters, but the limit of 650°C remains impassable for these steels. The new generation of steels with 12% chromium have been developed in order to overcome this barrier which has a direct impact on the performance of the boiler and reducing emissions. However, these steels are sensitive to oxidation at high temperature. La Fig.1 illustrates the different families of heat resistant steels.

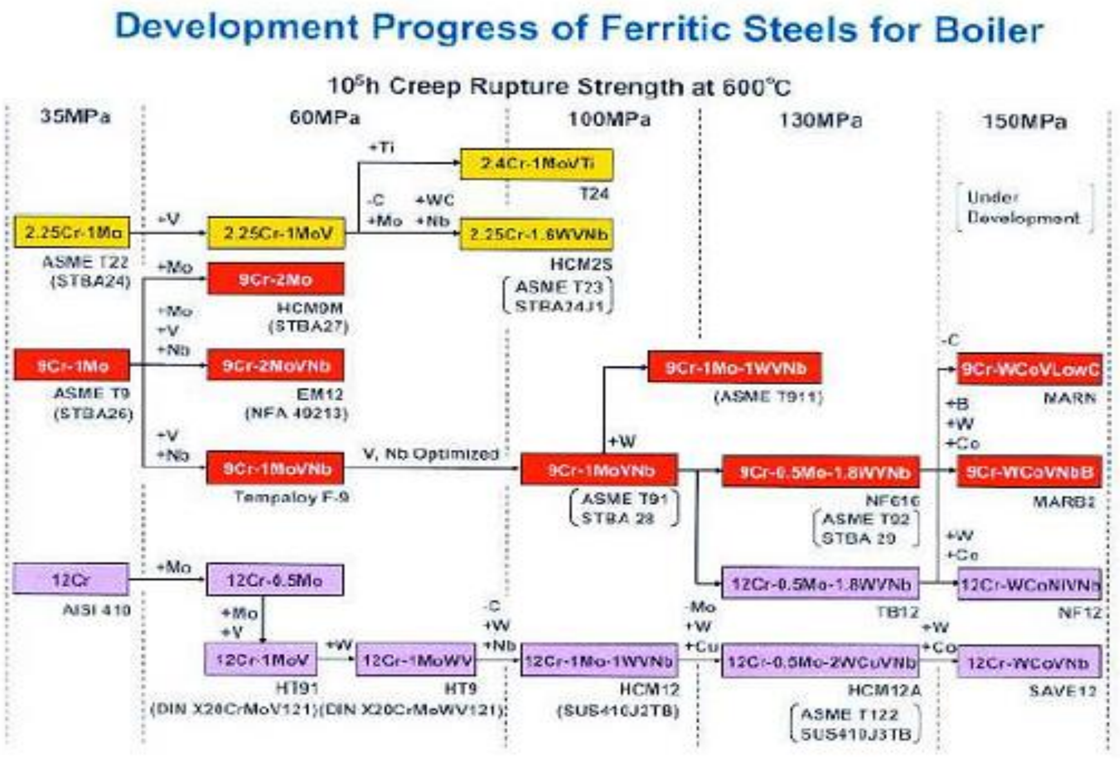

Figure 1. The different families of heat resistant steels [6]

2. The 9% Chromium Heat Resistant Steels

These steels are used in the manufacture of steam circulation lines in power plants. They allow high service temperature up to 650°C, but this value relative fall in the case of nuclear facilities (about 550° max). For the steel of this study, its name is linked with its strong alloying elements: chromium 9% chromium and molybdenum 1%. This magical combination entrusts the steel good resistance to creep and oxidation, and also good weldability.

The ECCC (European Collaborative Creep Committee) provides a life span of 500,000 hours T91 in moderate pressure and steam conditions[5].

A portion of a tube has been provided by the Tunisian Company of Electricity and Gas (STEG) to develop this research. Much attention was paid to the delivery of steel (not used) in order to control the initial conditions. Thus, in the following paragraphs, we are going illustrate the results of the characterization of steel (in its delivery condition) by various experimental means.

III. CARACTÉRISATION EXPÉRIMENTALE DU MATÉRIAU DE LIVRAISON

1. EDS Caracterisation

The EDS signal delivery material returns the following chemical composition:

Table 1: chemical composition of the steel delivery for elements whose content> 1%

Element	Wt %	At %
Mo	0.64	0.37
Cr	8.35	8.86
Fe	91.08	90.77
Total	100.00	100.00

The SEM-EDAX machine, used here, can not detect the elements of content below 1%. The elements chromium and molybdenum contribute to the formation of precipitates $M_{23}C_6$ (M = Cr or Mo) which are around the different types of joints and delay the movement of mobile dislocations. The impact of these elements on the microstructure of T91, is to allow the formation of MX precipitates, which constitute barriers against dislocation.

An EDS signal was recorded for steel T91 in its delivery condition.

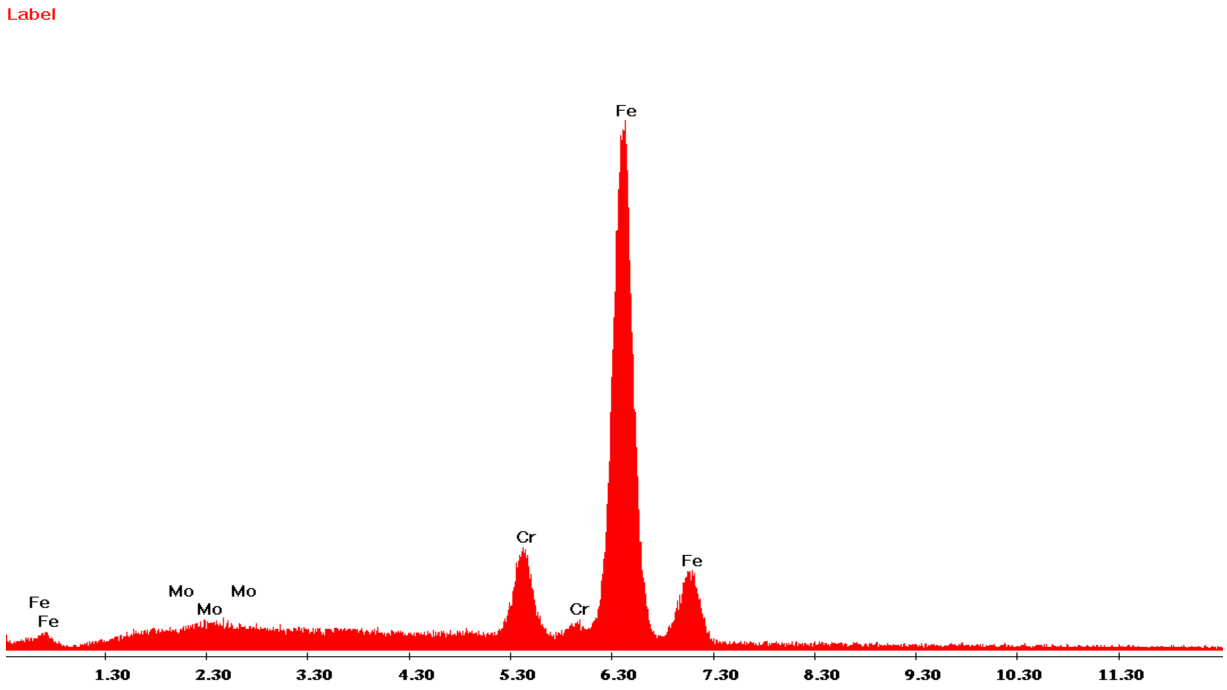

Figure 2. EDS Signal of the T91 in the as delivery condition

2. SEM Observations

The martensite matrix of T91 is shown in Fig.3. The size of the austenitic grain (PAGB or "Prior austenitic grain boundary" in red outline) is about 45 um in equivalent diameter. IT is divided into packets (orange outline), itself is split into block parallel laths (green outline).

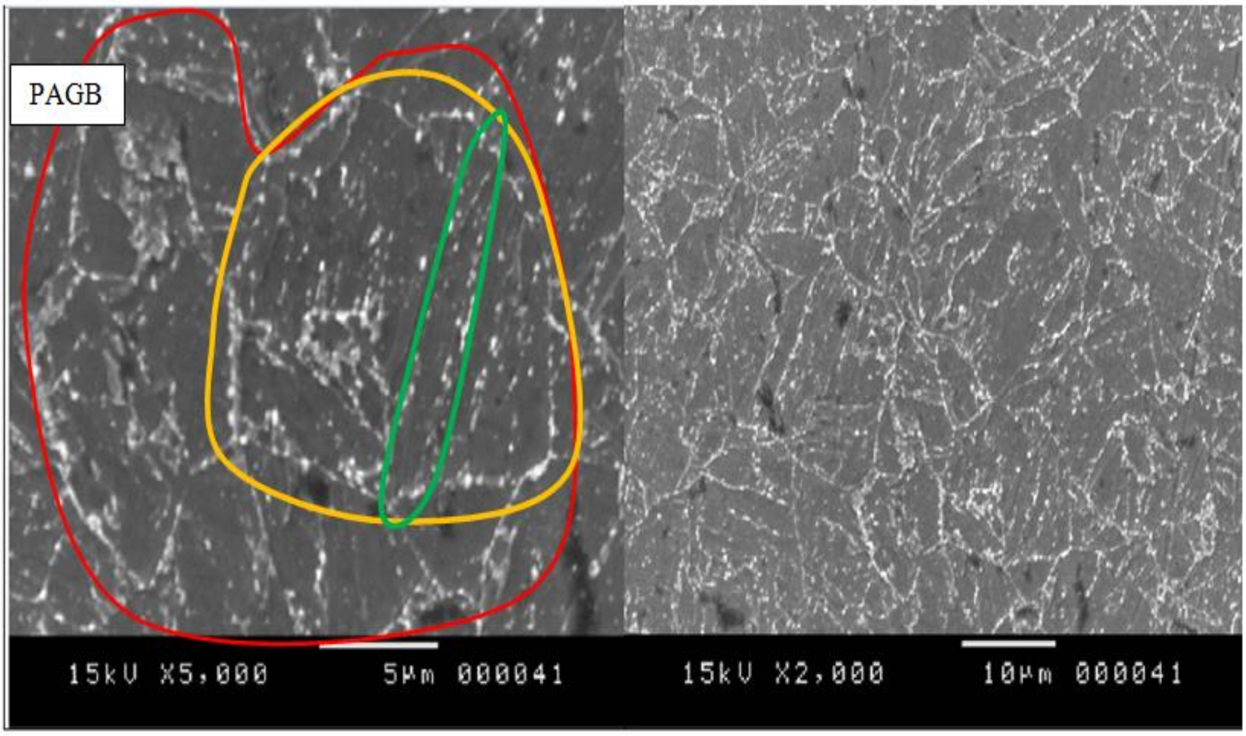

Figure 3. SEM Observations of the T91 in the as delivery condition

The distribution and the average size of the precipitates is observed in Fig.4. The precipitate size ranges from 20 nm to about 500 nm. This distribution will be clearly illustrated in the following paragraph.

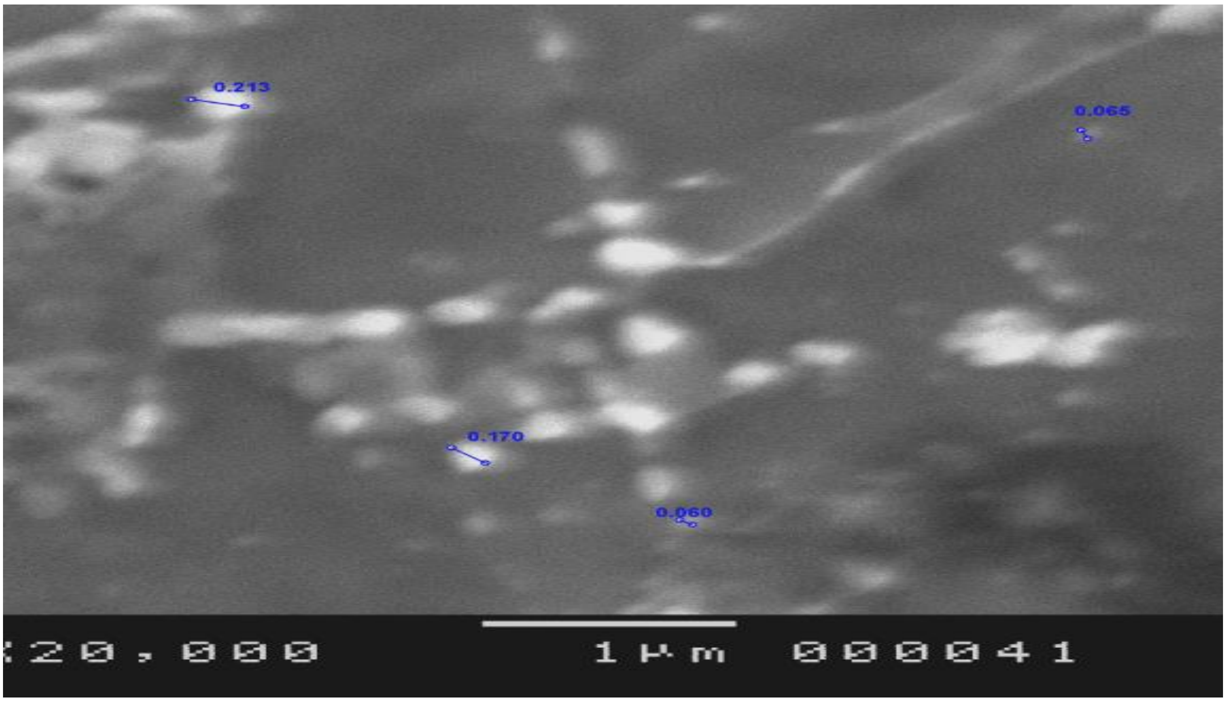

Figure 4. Illustration of precipitates

IV. MATERIAL CHARACTERIZATION

Four samples were analyzed, the heat exposure time are illustrated in the next Table.

Table1: Exposure time of the samples

Sample	1	2	3	4
Exposure time (hours) at 550°C	0	250	750	1250

1. Hardness

The hardness of three samples remains constant (224 Hv), confirming that no new phases nucleate due to creep without load.

2. SEM Observations

The microstructures of 4 samples are shown in Figure 5. Although quality of attack is poor for samples 3 and 4, as can be observed that the microstructure has not changed morphology.

But there is a slight increase in sizes of the precipitated portion of the third sample.

3. Analyse D'Image

The SEM pictures are analyzed by ImageJ Analysis to estimate the distribution of the precipitates according to their equivalent diameter (See Fig.6).

The equivalent diameter of the precipitates in the condition of delivery from 20 nm to 500 nm, with a concentration around 30 nm and 150 nm; this result is predictable since there are two morphologies precipitates.

After high-temperature retention, a slight increase of precipitates is observed: 200 nm maximum. This increase is not negligible, since the size of the $M_{23}C_6$ precipitates will increase to coalesce with each other, and in this case other morphologies may occur as phases of laves.

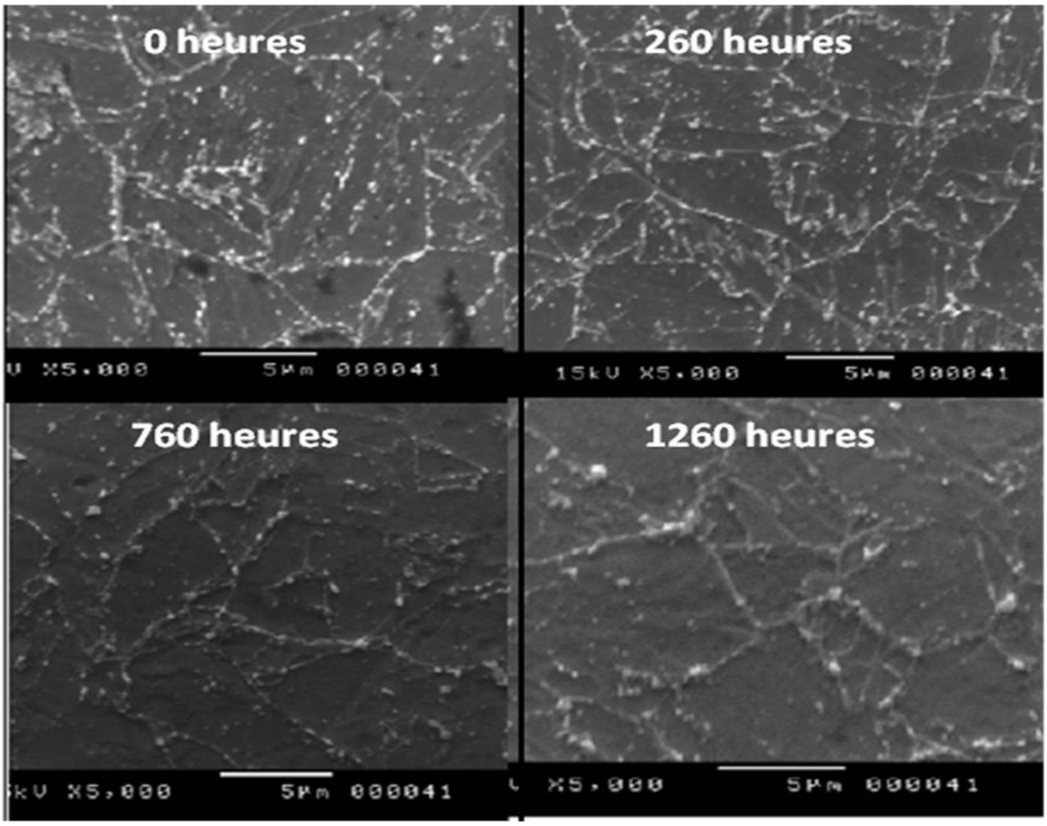

Figure 5. The changes in microstructure of the T91 depending on the holding time

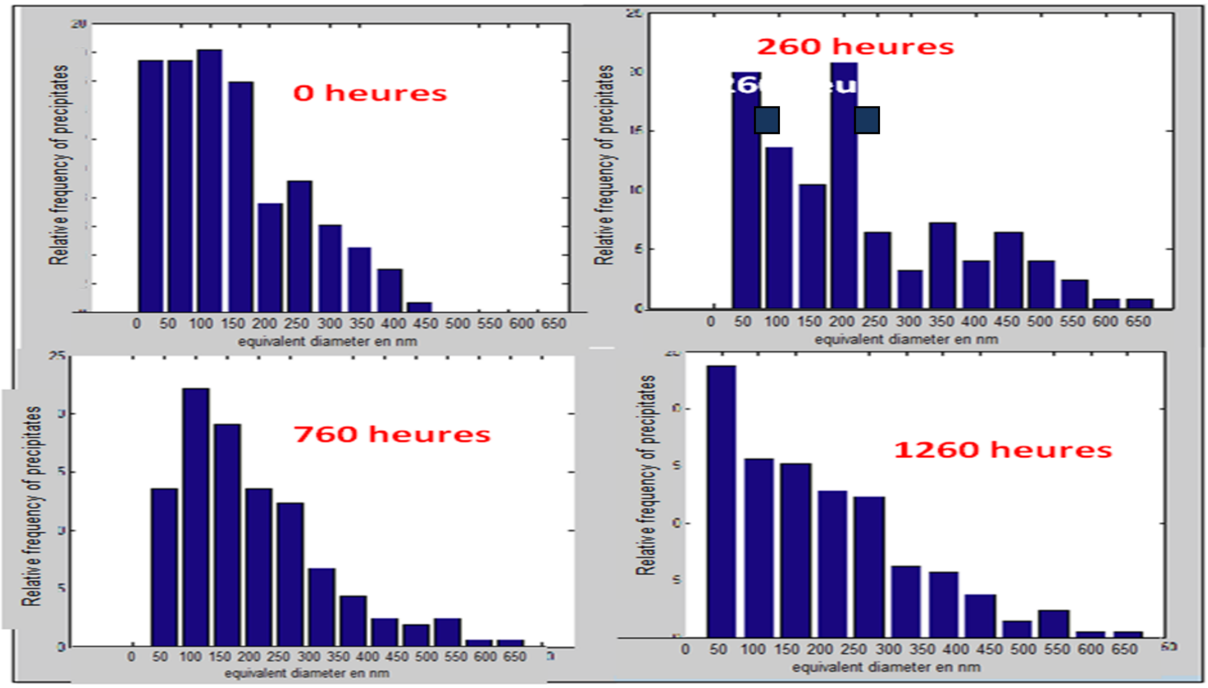

Figure 6. The relative frequency of precipitates in function of the holding time

V. CONCLUSIONS

Data collected, from samples exposed in the furnace, indicate a relative growth of precipitates over time. We must strictly follow this evolution in order to find the possible laws and the magnification mechanisms. Coarsening of precipitates is the phase that precedes the coalescence and absorption of the matrix elements which accelerates the flow of the material probably.

Load creep tests are being prepared, which will be presented in the following work to determine the contribution of the charge in the evolution of the degradation mechanisms of the material.

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